

REFLECTANCE EXPERIMENT LABORATORY (RELAB)

DESCRIPTION AND USER'S MANUAL

Spectroscopic data acquired in the laboratory provide the interpretive foundation upon which compositional information about unexplored or unsampled planetary surfaces is derived from remotely obtained reflectance spectra. The RELAB is supported by NASA as a multi-user spectroscopy facility, and laboratory time can be made available at no charge to investigators who are funded through NASA programs. RELAB has two operational spectrometers available to users: 1) a near-ultraviolet, visible, and near-infrared bidirectional spectrometer and 2) a near- and mid- infrared FT-IR spectrometer. The overall purpose of the design and operation of the RELAB bidirectional spectrometer is to obtain high precision, high spectral resolution, bidirectional reflectance spectra of earth and planetary materials. One of the key elements of its design is the ability to measure samples (hereafter referred to as **specimens**) using viewing geometries specified by the user. This allows investigators to simulate, under laboratory conditions, reflectance spectra obtained remotely (i.e., with spaceborne, telescopic, and airborne systems) as well as to investigate geometry dependent reflectance properties of geologic materials. The Thermo Nexus 870 FT-IR spectrometer is commonly operated in reflectance mode from 0.9 to 100 μm but can also be used for transmission measurements. An FTIR microscope (Thermo Continuum) is also attached to the bench FTIR and can be used in reflectance or transmission mode to measure spots as small as 50 μm on appropriate specimens. Use and scheduling of the RELAB is monitored by a 4-member advisory committee. NASA investigators should direct inquiries to the Science Manager or RELAB Operator.

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==> **RELAB Web Site: <http://www.planetary.brown.edu/relab/>**

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INSTRUMENT SPECIFICATIONS: UV-VIS-NIR BIDIRECTIONAL SPECTROMETER

Operating Spectral Range: Nominal (0.32 to 2.55 μm); Possible (0.3 to 2.6 μm)

<i>Mode</i>	<i>Range (μm)</i>	<i>Resolution (nm)</i>	<i>Grating</i>	<i>Detector</i>	<i>Light source</i>
A	0.30-0.42	< 1.7	1200	Photomultiplier	Xenon
B	0.40-0.85	< 1.7	1200	Photomultiplier	Halogen
C	0.60-1.80	< 3.4	600	LN2-cooled InSb-	Halogen
D	1.70-2.60	< 6.8	300	LN2-cooled InSb-	Halogen

Sampling Interval: 5 or 10 nm standard, 1 nm minimum.

Precision: Instrumental error is less than 1/4 % in reflectance.

Geometry: Bidirectional reflectance; Spectral goniometry.
Incident and reflected beam can each be varied from normal to 70°.
($-10 < i < 70$; $-70 < e < 70$).
Phase angle minimum 10°, maximum 140°.

Specimen: Particulate specimen or soil (reflectance):
Measured horizontal and uncovered (normally rotated slowly).
Optimal sample amount is 500 mg.
Specimen should be no smaller than 20 mg.
Specimen should be no larger than 10 g.
Chip or rock (reflectance):
Measured horizontal and uncovered on a height-adjustable stage.
Smallest reliable spot size is ~1.5 mm.
Maximum Specimen diameter is 10 cm (2.5 cm if it is to be spun).
Thin section (reflectance or transmittance):
Measured horizontal on halon (reflectance) or on an aperture (transmittance).
Regions to be measured should be no smaller than 1.5 mm.
Surface should be polished and clean (no carbon coating).

Reference Standard: Spectralon (SRS-99 by Labsphere).
Corrections based on the NBS calibration table and relative measurement to diffuse gold (at longer than 2 μm in wavelength) are applied automatically.

Light Source: Quartz halogen or Xenon lamp. Oriel monochromator.

Optics: Chopper, order sorting filters, front surface mirrors, depolarizer, apertures.

Typical Run Time: Measurements over a spectral range of 0.3 to 2.6 μm at 5 nm sampling interval take ~2 hours per specimen.

INSTRUMENT SPECIFICATIONS: Thermo Nexus 870 FT-IR SPECTROMETER

Operating Spectral Range: Nominal (1.0 to 50 μm) Possible (0.8 to 200 μm)

<i>Mode</i>	<i>Range (μm)</i>	<i>Light Source</i>	<i>Beam Splitter</i>	<i>Detector</i>
A	1.0– 4.7	Quartz	Si-on-CaF ₂	TE cooled DTGS
A*	0.9– 4.5	Quartz	Si-on-CaF ₂	LN ₂ cooled InSb
B	1.3 - 28	Glowbar	Ge-on-KBr	TE cooled DTGS
C	14.3-200	Glowbar	Solid substrate	DTGS

Resolution: 16, 8, 4, 2, 1 or 0.5 cm^{-1}

Default: 4 cm^{-1}

Geometry: Diffuse reflectance: Biconical, off-axis for Pike specimen holder
Transmission: normal incidence (collimated)

Sample: Particulate specimen or soil (reflectance):
Measured horizontal and uncovered in purged (H_2O , CO_2 free) environment.
Desired specimens amount is 50 - 500 mg.
Specimen should be no smaller than 20 mg.
More than 1 g is unnecessary.
Each sample should fill one of the dishes shown in Fig. 1.
Multiple specimens can be measured in sequence (same environment)
Chip (reflectance):
Measured horizontal and uncovered.
Regions to be measured should be no smaller than 1 mm.
Maximum size is 1 cm in height and 3 cm in diameter.
Thin section and single crystal (transmission):
Measured vertical, attached to an appropriate aperture board.
Regions to be measured should be no smaller than 1.5 mm.
Surface should be polished and clean (no carbon coating).

Reference Standard: Brushed diffuse gold (reflectance)
Blank (transmittance)

Additional Attachments: Pike AutoDiff Diffuse Reflectance Autosampler
Continuum Microscope system
Transmission mode currently operational
Reflectance mode (with 2-D capabilities) being tested.

Software: OMNIC, Atlas



Fig. 1. Black dishes for particular samples mounted on the multiple-spinning stage shown in Fig. 2.



Fig. 2. Multiple-spinning stage that allows up to five samples measured along with the standard material (Spectralon) while being spun at a rate of 1.5 seconds per rotation.

GENERAL OPERATIONS

Use of the RELAB should be initially arranged through the Science Manager. New Investigators must fill out the attached *User Registration Form*. All investigators should provide information for each specimen to be measured using the attached *Specimen Information Form* and *Compositional Analysis Form* if chemical or mineral composition is available. A completed *Measurement Request Form* must be provided for each set of specimens to be measured under a common measurement condition. This information is very important in order to maintain a useful and up-to-date data base of spectroscopic data.

Bidirectional Spectrometer:

Powders, chips, or slabs can be measured using the RELAB bidirectional spectrometer. Although specimen dishes come in various sizes, the smallest specimen that can be accurately handled is about 20 mg, while the optimum specimen size is about 500 mg. In all cases it is desirable for the specimens to be optically thick over the wavelength region of interest for reflectance measurements.

The standard specimen dish for the bidirectional spectrometer is aluminum coated with black teflon. Its reflectance is less than 3% throughout the entire spectral range of the bidirectional spectrometer.

For analysis of heterogeneous specimens, viewing optics include precision apertures for measurements from broad specimen areas up to 1 cm in diameter down to areas as small as 1 mm in diameter.

To initiate a data run the USER must supply and specify the following run parameters: (a) specimen name and properties; (b) angle of incidence (light source) and angle of emission (reflectance); (c) wavelength range (start and stop); and (d) sampling interval. Nominal settings are provided.

Based on these run parameters specified by the user, the RELAB Operator configures the system and performs all the data acquisition. A complete spectrum from 350 to 2550 nm at 5 nm sampling interval requires about 2 hours of laboratory run time per specimen, depending on specimen characteristics.

At the end of a run, values for the specimen signal, the standard signal, the offset, the ratio of specimen/halon (after offset subtraction), and the standard deviation of signal variance during integration on the specimen are recorded on disk in a file with all run parameters and text. These data files, each identified by a unique measurement ID, become part of the database collection. NBS calibration of halon is a multiplicative correction to the ratio data, producing absolute reflectance values for the specimen.

FT-IR Spectrometer:

A Nicolet Nexus 870 FTIR spectrometer is operational for particulate specimens using a PIKE AutoDiff multi-sample attachment. Infrared [off-axis] biconical reflectance spectra are normally produced relative to a gold standard. Infrared spectra may be scaled to and merged with bidirectional reflectance spectra to produce continuous spectra from 0.3 to 26 μm . The PIKE multi-sample biconical reflectance attachment is normally requested by outside users as the standard mode for mid-IR data. Use of the Nicolet Continuum microscope is available as well, but use of this instrument requires training and/or prior experience. Contact the RELAB science manager or operator for information on the use of the Continuum FTIR microscope.

USER INSTRUCTIONS

1) After the initial contact with the RELAB Science Manager to arrange use of the facility, users should contact the RELAB Operator directly to schedule specimens to be measured. Investigators should carefully select the most appropriate specimen preparation and RELAB configuration for their specific science requirements. If a user is uncertain of measurement strategy, he or she should contact the RELAB Operator or Science Manager for advice or suggestions. *[During periods of heavy use of the facility, requests for extensive numbers of spectra may be given lower priority in the measurement queue, therefore it is important to prioritize measurements in any large request.]*

2) Investigators (and/or their students) should fill out the attached **User Registration Form** with up-to-date information. Specimen IDs assigned at the RELAB use the investigators' three initials. **If you have published work using RELAB spectra, please also include a publication list.** These will be included in a public spectroscopy bibliography on the RELAB website. (It is unnecessary to send references already included.)

3) For *each set of specimens* to be measured under common conditions (same geometry, wavelength range, sample interval, etc), the investigator is requested to fill out the attached **Measurement Request Form**. It includes information to specify the type of measurement to be made in the RELAB.

- a) Applicable Specimens: Identify all the submitted specimen names which are to be measured under a common measuring condition described in the form, or write "All".
- b) Research Type: For accounting purposes, circle at least one category that accounts for support of this research, and please include NASA award number when applicable. The research type should reflect the NASA involvement of the PI/CoI or Sponsor.
- c) Wavelength Range and Sampling Interval or Resolution: Choose one of standard values or specify.
- d) Geometry: Incidence and emergence angles are measured from the vertical direction in positive and negative values of degrees (negative values are on the opposite site). For example, a geometry with source light at 30° inclination and detector at 20° inclination from the vertical direction would be (30, -20) which makes 50° phase angle. Phase angle must be 12° or larger for the Bidirectional Spectrometer and all measurements are currently made in the plane of scattering. Geometry for the FTIR Spectrometer is fixed, with broad conical incidence and reflectance at (30, -30), but out of the plane of scattering.
- e) Date Desired: Indicate the date spectral data need to be completed.
- f) Public Release Date: Specify when spectroscopic data on this specimen can be included in a public data base available to other investigators in digital form. Maximum period is three years. Default is immediate release.
- g) Data Transfer Preference: All files and data will be sent to the user electronically (e.g., e-mail).

4) For *each specimen* the investigator must fill out the attached **Specimen Information Form**. The information requested includes:

- a) Specimen Name & Description: Formal name or the investigator's name for the specimen (can be used in a database search) as well as a brief description of the sample
- b) Specimen Collection Location: The geographical site from which the specimen was obtained or 'synthetic' if it is not a natural material. If it is extraterrestrial, provide appropriate information - Meteorite fall/find location, Apollo lunar site, etc.
- c) Owner Name & Location: Owner and location where the specimen is likely to be (semi)permanently stored.
- d) Organic Type: Primarily inorganic, organic, or mixture (e.g., tar sands would be a mixture)
- e) Origin: Is the specimen natural, synthetic, both, or is it a synthetic sample derived or constructed from naturally occurring materials (e.g., a human made mixture of different natural minerals)?
- f) Texture: Powder, slab, coarse-grained sand, fine-grained soil, thin section, etc.
- g) Minimum and Maximum Particle Size: For particulate specimens, the maximum and minimum (if known) particle sizes in micrometers.
- h) Material Type and Names of Minerals, Rocks if applicable: Please mark the appropriate category and include names of minerals or rock types (sedimentary, igneous, metamorphic) when applicable.

5) Whenever available, *modal mineralogy* for the specimen and/or *chemical analysis* for either the bulk specimen or for individual mineral constituents should be provided on the **Compositional Analysis Form**. This important information will be stored in a separate file linked to the specimen. If such data are not currently available but are expected to be available in the future then please indicate this.

6) Specimens should be sent to the RELAB Operator with completed forms and any other necessary instructions. Although not necessary, any NASA investigator is welcome to visit the RELAB at Brown University to prepare their specimens for measurement in person and to use available data analysis tools. This should be prearranged to prevent schedule conflicts. A small office is available for visitors.

7) Data products are sent to the investigator (digital copy). Special requirements should be discussed with the RELAB Operator.

8) RELAB forms are downloadable in PDF form from our web site along with Microsoft Word files. Contact the RELAB Operator if there are any problems downloading the forms.

Specimen Information Form (One for each specimen)

Investigator: _____

Specimen Name & Description: _____

Specimen Collection Location: _____

Owner Name & Location: _____

Organic Type (mark one): Inorganic____ Organic____ Mixture____

Origin (mark one): Natural____ Natural & Synthetic____ Entirely Synthetic____
Synthetic From Natural (e.g., mixture of natural mineral powders)____ Flight Hardware____

Texture (e.g. particulate, non-particulate, pressed powder, etc.): _____

Particle Size: _____ μm
(Minimum) (Maximum)

Material Type (mark one): Mineral____ Rock____ Element____ Organic____ Amorphous____
Ice____ Unconsolidated Mixture____ Consolidated Mixture____ Single Particle____

Mineral Name(s) (if applicable): _____

Rock Type(s) (if applicable): _____

Is compositional information available? () Yes () No () Not currently, but will be acquired
(If yes, please also submit **Compositional Analysis Form.**)

Priority Level: _____ (used in **Measurement Request Form**)

* For RELAB use:

MM Analysis #: _____ Bulk Chem. #: _____

ID: _____ - _____ - _____ - _____ - _____

PI: _____ SI: _____ PO: _____

Compositional Analysis Form (One for each specimen if available)

Investigator: _____

Specimen Name: _____

Text (identify facility, method, and investigator performing analysis): _____

Chemical Analysis (wt%):

SiO ₂ : _____%	TiO ₂ : _____%	Al ₂ O ₃ : _____%	Cr ₂ O ₃ : _____%
V ₂ O ₃ : _____%	Fe ₂ O ₃ : _____%	FeO: _____%	CoO: _____%
NiO: _____%	MnO: _____%	MgO: _____%	ZnO: _____%
CaO: _____%	Na ₂ O: _____%	K ₂ O: _____%	P ₂ O ₅ : _____%
: _____%	: _____%	: _____%	: _____%

Include minor or trace elements information on a separate sheet.

Modal Mineralogy:

Mineral / Component	(wt/vol) %	Mineral / Component	(wt/vol) %
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

If available, provide chemical analysis for individual minerals on separate sheets.

* For RELAB use:

Specimen ID: _____

M Mineral #: _____

C Analysis #: _____

Measurement Request Form (One for each group or subgroup of specimens)

Investigator Name: _____

Applicable Specimens: _____

Research Type (circle at least one): Student NSF EW SSW HW Astronomy
Exobiology LDAP MDAP PSTAR DDAP NASA-Foreign SSERVI PDART
LARS CDAP NFDAP Active missions Mission planning Proposal preparation
Other: _____

NASA PI/CoI or Sponsor (if different from Investigator) : _____

Bidirectional Spectrometer (* Standard parameters)

Wavelength Range: ()* 0.32-2.55 μm () 0.3-2.6 μm () Other _____
Sampling Interval: ()* 10 nm () 5 nm () Other _____
Geometry (incidence, emergence angles): ()* (30, 0) () Other _____

FT-IR Spectrometer (* Standard parameters)

Wavelength Range: () 1-4 μm ()* 2-25 μm () 17-50 μm () Other _____
Wavenumber Resolution: () 1 cm^{-1} () 2 cm^{-1} ()* 4 cm^{-1} () Other _____

Schedule (* Maximum and default)

Date desired for the priority levels specified in the Specimen Information Forms

Priority Level: _____

Date Desired: _____

Public Release Date: () Immediate () 1 Year ()* 2 Years